



(11) (A) No. 1 173 604

(45) ISSUED 840904

(52) CLASS 9-16

(51) INT. CL. D21C 1/08<sup>3</sup>

(19) (CA) **CANADIAN PATENT** (12)

(54) Production of Chemimechanical Pulp

(72) Prusas, Zenon C.,  
U.S.A.

(73) Granted to Mead Corporation (The)  
U.S.A.

(21) APPLICATION No.	394,564
(22) FILED	820120
(30) PRIORITY DATE	U.S.A. (233,415) 810211

No. OF CLAIMS 30 - NO DRAWING

Canada

### Abstract of the Disclosure

5. A process is provided for the production of chemimechanical pulp in the absence of silicate stabilizers. This chemimechanical pulp is characterized by (1) a pulp yield of at least 80% based on dry chips, (2) strength properties as shown by a combination of tear, burst and tensile properties which are superior to refiner mechanical pulp, and, (3) bleaching properties as shown by brightness properties which are superior to both refiner mechanical pulp and cold soda pulp. 10 The chemimechanical pulp is prepared by impregnating hardwood chips with an alkaline peroxide liquor. The alkaline peroxide liquor is an aqueous solution containing about 0.5% to about 4% hydrogen peroxide and about 2.5% to about 10% sodium hydroxide based on dry chips. The 15 impregnated chips have a pH greater than 7.5. The thus impregnated wood chips are then held for about 3 minutes to about 3 hours to soften and bleach the chips after which the chips are acidified to a pH of 7.5 or below by the addition of an acid material and thereafter defibrated by passing the acidified chips through refining apparatus operated at atmospheric pressure. The pulp produced by this defibration is recovered from the refining apparatus. 20

THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE  
PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

- 1) A process for the production of chemimechanical pulp in the  
absence of silicate stabilizers, said pulp being characterized  
by a pulp yield of at least 80% based on dry wood chips; strength  
properties as shown by a combination of tear, burst and tensile  
properties which are superior to refiner mechanical pulps; and  
bleaching properties as shown by brightness properties which are  
superior to both refiner mechanical pulp and cold soda pulp;  
said process comprising the steps of:
- a) impregnating hardwood chips with an alkaline peroxide liquor,  
said alkaline peroxide liquor being an aqueous solution  
characterized by containing about 0.5% to about 4% hydrogen  
peroxide, and about 2.5% to about 10% sodium hydroxide both  
based on bone dry wood chips, said impregnated wood chips  
having a pH greater than 7.5;
  - b) holding said impregnated chips for from about 3 minutes to  
about 3 hours to soften and bleach said chips;
  - c) acidifying said chips to a pH of 7.5 or below by addition  
of an acid material;
  - d) defibrating said acidified bleached chips by passing said  
acidified bleached chips through refining apparatus, said  
refining apparatus being operated at atmospheric pressure,  
said defibrating producing wood pulp; and
  - e) recovering said wood pulp from said refining apparatus.

- 2) The pulping process of claim 1, wherein said wood chips are subjected to steam prior to said impregnation step.
- 3) The pulping process of claim 1, wherein said wood chips are impregnated by compressing said chips and allowing said compressed chips to expand in said alkaline peroxide liquor.
- 4) The pulping process of claim 1, wherein said alkaline peroxide liquor contains about 1% to about 3.0% hydrogen peroxide.
- 5) The pulping process of claim 1, wherein said alkaline peroxide liquor contains about 4% to about 8% sodium hydroxide.
- 5 6) The pulping process of claim 1, wherein said alkaline peroxide liquor additionally contains a complexing agent taken from the group consisting of diethylenetriaminepentacetic acid, 2-hydroxyethylethylenediaminetriacetic acid, ethylenediaminetetracetic acid, diethylenetriaminepenta(methylenephosphonic) acid, their alkali metal salts and combinations thereof.
- 7) The pulping process of claim 1, wherein said acid material is selected from the group consisting of sulfuric acid, sodium bisulfite, sulfur dioxide, acetic acid, phosphoric acid, hydrochloric acid, and combinations thereof.

- 8) The pulping process of claim 1, wherein said pH of said acidified bleached chips is about 6 to about 7.
- 9) The pulping process of claim 1, wherein said acidified bleached chips have a consistency of about 20% to about 30% during the defibrating step.
- 10) The pulping process of claim 1, wherein said wood pulp is subjected to one or more additional refining steps.
- 11) The pulping process of claim 1, wherein said wood pulp is subjected to one or more additional bleaching steps.
- 12) The pulping process of claim 1, wherein said impregnated chips are bleached until white in color.
- 13) The pulping process of claim 1, wherein said refining apparatus comprises a disc refiner.
- 14) The pulping process of claim 2, wherein said impregnated chips are held at a temperature of about 100°F to about 212°F for a period of 3 minutes to 3 hours prior to the acidification step.

- 15) The pulping process of claim 1, wherein said wood chips are aspen chips.
- 5 16) A process for the production of chemimechanical pulp in the absence of silicate stabilizers, said pulp being characterized by a pulp yield of at least 80% based on dry wood chips; strength properties as shown by a combination of tear, burst and tensile properties which are superior to refiner mechanical pulps; and  
bleaching properties as shown by brightness properties which are superior to both refiner mechanical pulp and cold soda pulp; said process comprising the steps of:
- 10 a) steaming hardwood chips to soften said chips;  
b) compressing said chips by means of a screw press;  
c) impregnating said chips with an alkaline peroxide liquor by allowing said compressed chips to expand in said alkaline peroxide liquor, said alkaline peroxide liquor being an aqueous solution characterized by containing about 0.5%  
15 to about 4% hydrogen peroxide, about 2.5% to about 10% sodium hydroxide both based on bone dry wood chips and additionally containing an organic complexing agent, said impregnated wood chips having a pH greater than 7.5;  
d) holding said impregnated chips at a temperature of about  
20 100°F to about 212°F for a period of 3 minutes to 3 hours to further soften and bleach said chips;  
e) acidifying said chips to a pH of 7.5 or below by addition of an acid material, said acid material being selected from the group consisting of sulfuric acid, sodium bisulfite,  
25 sulfur dioxide, acetic acid, phosphoric acid, hydrochloric acid, and combinations thereof;  
f) defibrating said acidified bleached chips by passing said acidified bleached chips through refining apparatus, said refining apparatus comprising a disc refiner operated at  
30 atmospheric pressure, said defibrating producing wood pulp; and

- g) recovering said wood pulp from said refining apparatus.
- 17) The pulping process of claim 16, wherein said alkaline peroxide liquor contains about 1% to about 3.0% hydrogen peroxide.
- 18) The pulping process of claim 16, wherein said alkaline peroxide liquor contains about 4% to about 8% sodium hydroxide.
- 19) The pulping process of claim 16, wherein said organic complexing agent is selected from the group consisting of diethylenetriamine-pentacetic acid, 2-hydroxyethylethylenediaminetriacetic acid, ethylenediaminetetraacetic acid, diethylenetriaminepenta(methylene-phosphonic) acid, their alkali metal salts and combinations thereof.
- 20) The pulping process of claim 16, wherein said pH of said acidified bleached chips is about 6 to about 7.
- 21) The pulping process of claim 16, wherein said acidified bleached chips have a consistency of about 20% to about 30% during said defibrating step.
- 22) The pulping process of claim 16, wherein said wood pulp is subjected to one or more additional refining steps.

- 23) The pulping process of claim 16, wherein said wood pulp is subjected to one or more additional bleaching steps.
- 24) The pulping process of claim 16, wherein said wood chips are aspen chips.
- 25) A process for the production of chemimechanical pulp in the absence of silicate stabilizers, said pulp being characterized by a pulp yield of at least 80% based on dry wood chips; strength properties as shown by a combination of tear, burst and tensile properties which are superior to refiner mechanical pulps; and bleaching properties as shown by brightness properties which are superior to both refiner mechanical pulp and cold soda pulp; said process comprising the steps of:
- a) steaming aspen chips to soften said chips;
  - b) compressing said chips by means of a screw press;
  - c) impregnating said chips with an alkaline peroxide liquor by allowing said compressed chips to expand in said alkaline peroxide liquor, said alkaline peroxide liquor being an aqueous solution characterized by containing about 1% to about 3% hydrogen peroxide, about 4% to about 8% sodium hydroxide both based on bone dry wood chips and additionally containing an organic complexing agent selected from the group consisting of diethylenetriaminepentacetic acid, 2-hydroxyethylethylenediaminetriacetic acid, ethylenediaminetetraacetic acid, diethylenetriaminepenta(methylenephosphonic) acid, their alkali metal salts and combinations thereof, said impregnated wood chips having a pH greater than 7.5;
  - d) holding said impregnated chips at a temperature of about 100°F to about 212°F for a period of 3 minutes to 3 hours to further soften and bleach said chips;



- e) acidifying said chips to a pH of about 6 to about 7 by addition of an acid material, said acid material being selected from the group consisting of sulfuric acid, sodium bisulfite, sulfur dioxide, acetic acid, phosphoric acid, hydrochloric acid, and combinations thereof;
  - f) defibrating said acidified bleached chips by passing said acidified bleached chips through refining apparatus, said refining apparatus comprising a disc refiner operated at atmospheric pressure, said defibrating producing wood pulp; and
  - g) recovering said wood pulp from said refining apparatus.
- 26) The pulping process of claim 25, wherein said wood pulp is subjected to one or more additional refining steps.
- 27) The pulping process of claim 25, wherein said wood pulp is subjected to one or more additional bleaching steps.
- 28) The pulping process of claim 16, wherein said acidified bleached chips have a consistency of in excess of about 8% during said defibrating step.
- 29) The pulping process of claim 16, wherein said acidified bleached chips have a consistency of in excess of about 10% during said defibrating step.
- 30) The pulping process of claim 16, wherein said acidified bleached chips have a consistency of in excess of about 12% during said defibrating step.



Background of the Invention

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This invention relates to a process for the production of a high-yield, high-strength bleached chemimechanical pulp from hardwood chips using a treatment with an alkaline peroxide liquor prior to defibration of the chips.



The use of bleached hardwood chemical pulps in the manufacture of papers, in particular printing papers, has increased in recent years. This increase in demand for this relatively short fiber pulp has been due to the fact that this pulp, which is used in conjunction with long fiber pulps, has relatively high strength, fold, tear, burst and tensile, as compared to previously used refiner pulps, and thereby contributes increased strength to the finished paper. Manufacture of such hardwood chemical pulps by the kraft process has the disadvantage of malodorous emissions from the pulping process. The odor contributes heavily to pollution of the atmosphere. An additional disadvantage of the hardwood chemical pulping process is the relatively low yield, being in the range of 40% to 50% on the dry chips depending on the wood and the processing steps. A further disadvantage is the high capital cost of producing the hardwood chemical pulps.

Using the process of the instant invention, a relatively high-strength, high-yield pulp can be produced. This pulp can be used to essentially replace bleached hardwood chemical pulps in various types of white papers without the aforementioned disadvantages in the manufacture of the bleached hardwood chemical pulps. The process of this invention comprises the steps of: (1) impregnating wood chips with an alkaline peroxide liquor of high alkalinity, (2) holding the impregnated chips for a time sufficient to bleach the impregnated chips, (3) acidifying the bleached chips, and thereafter, (4) defiberizing the chips by passing through a refining apparatus under atmospheric conditions. A discussion of the prior art relevant to the instant invention is as follows:

U. S. Patent No. 3,023,140, issued February 27, 1962, to Textor, and U. S. Patent No. 3,069,309, issued December 18, 1962, to Fennell, disclose refiner bleaching processes wherein bleaching of wood chips is accomplished by treatment at the refiner with alkaline peroxide liquors. In each process, sodium silicate and magnesium sulfate are added to the bleaching liquor and the alkalinity of the liquor is low, thus minimizing any pulping effect the liquor might have on the chips.

U. S. Patent No. 2,958,622, issued November 1, 1960, to Sparrow, discloses a pulping process for cellulose containing materials, such as wood shavings, using an alkaline oxidizing bleaching agent, such as a hypochlorite solution, for treating the wood shavings prior to mechanical disintegration of same. Sodium peroxide is suggested as an alternate oxidizing bleaching agent. U. S. Patent No. 3,016,324, issued January 9, 1962, to Textor, discloses a pulping process and apparatus for wood chips wherein the wood chips may be impregnated with sodium sulfite liquor by squeezing the chips in a screw press and spraying the chips emerging from the press with sulfite liquor. Sodium peroxide, together with additives such as sodium silicate and epsom salts, is disclosed as an alternative to the sodium sulfite.

French Patent 1,238,412, granted July 4, 1960, discloses the impregnation of wood chips with alkaline peroxide plus a stabilizer, such as an alkali silicate, for not more than an hour during which time the temperature of the chips may be raised to 100°C, defibering the chips and storing the defibrated chips for two minutes to two hours before refining and washing. The alkaline peroxide is sodium peroxide.

U. S. Patent No. 3,558,428, issued January 26, 1971, to Asplund, discloses a chemimechanical pulping process wherein lignocellulose material is impregnated with alkaline hydroxides or alkaline carbonates at a temperature below 100°C. The impregnated chips are cooked in a digester in an atmosphere of saturated steam at a temperature above 100°C for a time period ranging from 1 to 20 minutes. During this cooking period, sulfur dioxide SO<sub>2</sub> gas is added with the steam to react with the alkaline impregnating materials to form alkali sulfite compounds. The cooked chips are then defibrated under the pressure and temperature prevailing in the digester.

U. S. Patent No. 4,187,181, issued February 5, 1980, to Ahrel, discloses a process of producing bleached mechanical pulp of increased brightness and light scattering properties. In this process, screw pressed-wood-chips-are-impregnated-with-an-alkaline-peroxide-solution and surplus impregnating solution is removed by compression of the chips. The impregnated chips are introduced into a pressure vessel and ground between a pair of rotating discs. This prior art discloses

that in the absence of peroxide, the optical properties of the fibers deteriorates by storage of the alkalized raw fibers. Thus, temperature and time of retention prior to defibration and temperature of defibration must be controlled or restricted to insure retention of peroxide before and during defibration. To do this, compressed air is added to the pressure vessel to maintain the chips at a temperature below 90°C while the chips are being passed to the grinding zone. The impregnating solution is a weakly alkaline solution and may contain 0.5% to 1.5% sodium hydroxide, based on dry wood. The impregnating solution may also contain, if necessary, the usual chemical adjuvants, such as; e.g., water glass (sodium silicate), a magnesium salt or a complex forming substance.

As can be noted from the cited prior art, the alkaline peroxide liquors for bleaching and pulping normally contain an alkali silicate, generally sodium silicate, to stabilize the peroxide. Using such silicates, silica will gradually build up on the refiner plates and drastically shortens plate life. Also silica will deposit in the evaporator system during known chemical recovery processes. The silica is difficult to remove and, thus, adds to the cost of the pulping process. Similar deposition of difficult-to-remove silica deposits also occurs to a lesser extent in the furnace where the concentrated liquor, which contains waste wood products from the pulping process, is burned. As a result of this deposition of silica, pulping effluents containing sodium silicate are difficult to dispose of and sometimes are sent to the sewer, thus contaminating rivers and streams below the effluent outfall. Alternatively, such effluents are subjected to costly treatments which either remove and/or destroy the organic content of the effluent.

Further, it is known from the cold soda process that under certain conditions of high alkalinity, hardwood chips and/or hardwood pulp will often turn yellow to light orange. Subsequent bleaching of the discolored pulp to a higher brightness requires inordinate amounts of bleaching chemicals and time. It has been observed by the inventor that development of this difficult-to-bleach color in alkaline peroxide

pulping can occur due, in part, to the high alkalinity of the impregnating liquor if the peroxide is depleted prior to or during defibration in a disc refiner. Localized temperatures in the refiner may be as high as 320°F. A combination of: (1) high alkalinity of the liquor, (2) absence of peroxide, and (3) high temperature of the impregnated chips and pulp are the conditions conducive to formation of the yellow to orange color. Contaminating metal ions, in particular iron and manganese, function as catalysts to decompose the peroxide, thereby causing formation of objectionable color. The source of the contaminating metal ions may be the wood chips, water, or it may be from the manufacturing apparatus.

It is an object of this invention to provide a process of chemimechanical pulping using alkaline peroxide liquor, whereby a bleached hardwood pulp of high-strength, high-yield and high-brightness can be produced.

It is a further object of this invention to provide a chemimechanical pulping process wherein the pulping chemicals can be readily recovered and waste wood products can be eliminated from mill effluents at minimal cost.

#### Statement of the Invention

A process is provided for the production of chemimechanical pulp in the absence of silicate stabilizers. This chemimechanical pulp is characterized by (1) a pulp yield of at least 80% based on dry chips, (2) strength properties as shown by a combination of tear, burst and tensile properties which are superior to refiner mechanical pulp, and, (3) bleaching properties as shown by brightness properties which are superior to both refiner mechanical pulp and cold soda pulp. The chemimechanical pulp is prepared by impregnating hardwood chips with an alkaline peroxide liquor. The alkaline peroxide liquor is an aqueous solution containing about 0.5% to about 4% hydrogen peroxide and about 2.5% to about 10% sodium hydroxide based on dry chips. The impregnated

chips have a pH greater than 7.5. The thus impregnated wood chips are then held for about 3 minutes to about 3 hours to soften and bleach the chips after which the chips are acidified to a pH of 7.5 or below by the addition of an acid material and thereafter defibrated by passing the acidified chips through refining apparatus operated at atmospheric pressure. The pulp produced by this defibration is recovered from the refining apparatus.

### Detailed Description of the Invention

The process of this invention can be used in pulping cellulose material, in particular hardwood chips such as aspen, oak, maple and birch chips. Excellent results have been obtained on aspen chips. The resulting bleached pulp can be used as a substitute for the more expensive bleached chemical pulps. It is particularly adaptable for use as the short fiber in printing papers. This pulp could also be used as a component of, for example, tissue or absorbent papers or any other papers where hardwood chemical pulps are presently being used.

The impregnating step uses an alkaline peroxide liquor which contains from about 0.5% to about 4%, by weight, preferably about 1% to about 3%, hydrogen peroxide based on the amount of dried wood chips. The amount of hydrogen peroxide needed for bleaching would vary, depending on the degree of bleaching desired and the type and age of wood chips used. Oak chips, for example, would require a higher amount of peroxide than, for example, aspen chips. Above 4% peroxide can be used, but any benefits from the added peroxide would be minimal.

The alkaline peroxide liquor also contains about 2.5% to about 10%, by weight, preferably about 4% to about 8%, sodium hydroxide based on the amount of dried wood chips. Above 10% sodium hydroxide may be used, but the benefits from the added sodium hydroxide would be minimal.

Stoichiometric amounts of sodium peroxide can be substituted for all or part of the hydrogen peroxide and all or part of the sodium hydroxide, if desired.

5       The alkaline peroxide liquor may additionally contain a chelating or otherwise complexing agent to control contamination with metal ions, particularly iron and manganese ions. Preferred agents are organic complexing agents, such as diethylenetriaminepentacetic acid (DTPA), 2-hydroxyethylethylenediaminetriacetic acid (HEDTA), ethylenediaminetetracetic acid (EDTA), diethylenetriaminepenta(methylenephosphonic) acid, their alkali metal salts, and combinations thereof. The use of about 0.5% DTPA on a dried wood chip basis has been found to give beneficial results. If recovery of the sodium hydroxide is desirable, inorganic complexing agents, such as, in particular sodium silicate, are to be avoided. Sodium silicate causes a gradual silica buildup on the recovery apparatus, primarily on the evaporators. The silica is difficult, if not impossible, to remove. Thus adding to the cost of chemical recovery. Sodium silicate also is known to precipitate silicate on the refiner plates during defibration, resulting in reduced plate life.

20       In a preferred form of the invention, the wood chips are compressed to remove some of the chip water prior to the impregnating step and then allowed to expand while immersed in the alkaline peroxide liquor. This compressing and impregnation is conveniently accomplished using a Bauer Impressafiner which is disclosed and described in U. S. Patent No. 2,975,096 (1976) to Gínaven et al. Alternately, impregnation may be done using the PREX (pressure-expansion) system developed by Sands Defibrator AB, Stockholm, Sweden.

25       Impregnation of the chips may be further facilitated by pretreating the wood chips with steam prior to compressing and/or the chips may be impregnated in two stages with an optical dewatering (compressing) step in between the two impregnating stages, if desired. The manner of impregnation or apparatus for accomplishing the same are not critical to the practice of this invention.



The impregnated wood chips are held for a period of time sufficient to bleach the chips until they are white in color. This can occur over a period of time from about 3 minutes to about 3 hours, depending on the temperature, size and type of wood chips. Oak chips, for example, would take longer to bleach than the less dense hardwood chips, for example, aspen chips. Aspen chips are preferably held for about 10 minutes to about 20 minutes at a temperature of about 120°F to about 160°F. During this time, the relatively high alkalinity of the alkaline peroxide softens the wood chips and thus enables the fibers to more easily separate during the subsequent defibrating step.

The impregnated chips are then neutralized or made slightly acid by the addition of an acid material, such as inorganic or organic acids or acid salts. Typical acid materials are sulfuric acid, sodium bisulfite, sulfur dioxide, acetic acid, phosphoric acid and hydrochloric acid. Combinations of these acid materials may be used, if desired. The pH of the acidified bleached chips should be about 7.5 or below, preferably about 6 to about 7.

The acidified bleached chips are defibrated in a refining apparatus, preferably a disc refiner. The disc refiner is operated at atmospheric pressure. A typical refiner, which can be used in the process of this invention, is a C-E Bauer Double Disc Refiner. Control of temperature of the refining is not necessary for the practice of this invention. Temperatures in the refiner are in excess of 212°F.

The consistency of the acidified bleached chips during the defibrating step is preferably about 20% to about 30%, but lower consistencies in excess of about 8% such as consistencies in excess of about 10% or in excess of about 12% and higher consistencies up to 35% can be tolerated.

If desired, the defibrated bleached pulp may be further refined in one or more known refining steps. If higher brightness is desirable, the pulp may be additionally bleached by one or more known bleaching steps.

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~~A comparison of pulps produced by the process of this invention~~  
with a typical refiner mechanical pulp and a typical cold soda pulp as controls disclosed an unexpected combination of improvement in brightness, opacity and strength values of the instant pulps over the control pulps. Aspen chips were used in these comparative pulping processes. The magnitude of the improvements was also unexpected.

X

5 The instant pulps were shown to have an increase of about 5 to 15 points in brightness over the refiner mechanical pulp and an increase of about 20 to 30 points in brightness over the soda pulp. A two-stage bleaching of the cold soda pulp after the pulping process only increased the brightness of the soda pulp by about 16 points, to about 64 brightness which is too low for most white printing papers. Standard hand-sheets produced using the instant pulps were shown to have burst values of about 2 to 5 times, tear values of about double, tensile values of about 2 to 3 times and fold values of about 10 to 50 times as compared to the typical refiner mechanical pulp. In general, the strength values of the instant pulps were comparable to those of cold soda pulp. Optical scattering values of the instant pulps showed about a 50% to 60% increase in optical scattering over a typical cold soda pulp.

10 In the practice of this invention, yields of pulp on wood chips were in the order of 85% to 90% based on dried wood chips. Color reversion on aging was minimal. Where the chips were acidified before the defibrating step, the yellow to light orange, difficult-to-bleach color did not develop. In several instances, where the impregnated wood chips were not neutralized before the refining step, the yellow color developed in the refined pulp. This was particularly true if iron and/or manganese were present.

15 Spent alkaline peroxide liquor can be removed from the system by washing and/or compressing the defibered pulp. Excess water may be removed by evaporation from the spent liquor with subsequent recovery of the sodium values, and sulfur values if present, from the concentrate. Alternatively, the spent liquors may be used, in part, in the preparation of fresh alkaline peroxide liquors or the spent liquors may be used as wash water in a normal kraft pulping process with the subsequent recovery of sodium and sulfur values in the kraft recovery system.

25 30 The following examples further illustrate the preferred embodiments of this invention and the advantages obtained thereby without limiting the scope of the invention. All tests were performed according to TAPPI standard testing procedures.

**SUBSTITUTE**  
***REMPLACEMENT***

**SECTION is not Present**  
***Cette Section est Absente***

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Examples 1 and 2

Two runs were made according to the teachings of this invention using 560-GS Bauer Impressafiner and C-E Bauer 401 Atmospheric Double Disc Refiner. The procedure was as follows. About 200 lbs. of aspen chips (bone dry basis) were presteamed for about 10 minutes with low pressure steam. Presteaming was done in a hopper located above the Impressafiner. Presteamed chips were then fed into the Impressafiner having a capacity of about 60 tpd bone-dry chips. As noted supra, the Impressafiner is a screw press with an attached impregnation vessel. Impregnation liquor containing sodium hydroxide and hydrogen peroxide was continuously supplied to this impregnation vessel. An overflow was provided for excess liquor. Compressed chips were allowed to expand in this impregnation vessel and they absorbed the liquor while expanding.

The impregnated chips were allowed to drain on the floor for about 25-30 minutes before refining them in 401 DD Bauer refiner. Chips became whiter during this storage. Twenty (20) lbs. of these chips, bone-dry basis, were used for each refiner run. SO<sub>2</sub> dissolved in water was injected into the eye of the refiner during the first pass through the refiner. The quantity of acid was regulated according to the pH of pulp leaving the refiner. An attempt was made to keep the pH below 7. Actual pH levels were 5.6 for Example 1 and 5.2 for Example 2. In Example 2, material refined in the first pass was additionally refined during the second pass through the refiner. Example 1 represents one pass refining operation.

Examples 3 and 4

Two prior art pulps were produced for comparison with pulps from Examples 1 and 2, using the same source of chips and the same equipment as in Examples 1 and 2. These were a RMP (refiner mechanical pulp) and a cold soda pulp. In the production of RMP pulp (Example 3) there was no chip presteaming or impregnation prior to defibrating and

refining. Chips as received were fed directly into the Bauer 401 refiner in 20 lb. batches and the resulting pulp was additionally refined in the second pass through the same refiner. Procedures in the production of cold soda pulp were very similar to those of Example 2, except that different impregnation chemicals (caustic soda only) were used and there was no acidification of chips prior to defibrating.

Pulp testing results are shown in the following table (Table I).

TABLE I

Process	Chemi-Mechanical		Prior Art	
			RMP	Cold Soda
Example No.	1	2	3	4
Number of refiner passes	1	2	2	2
HPD/T	N/A	41.2	82.5	35.5
<u>Chemicals Applied on Chips (bone-dry basis), %:</u>				
NaOH	5.0	5.0	0	7.4
H <sub>2</sub> O <sub>2</sub>	1.9	1.8	0	0
DTPA	0.5	0.5	0	0
<u>Pulp Properties:</u>				
Brightness, Initial	76.5	74.4	63.3	48.1
Brightness, Aged *	75.0	72.8	61.9	47.6
C.S. Freeness, ml	205	95	90	135
Bulk, cc/g	2.33	2.27	3.08	1.76
Tear Factor	75.3	69.9	36.6	71.9
Burst Factor	20.0	23.8	7.1	29.5
Tensile, b.l.m.	4435	4680	2080	6100
MIT Fold	9	8	1	30
TAPPI Opacity	81.1	81.5	95.6	86.9
Optical Scattering, S	.0417	.0425	.0706	.0274

\* Aged for 1 hour in oven at 105°C.

5 Chemimechanical pulps made according to the teachings of this invention were brighter by about 12 points and much stronger than the conventional RMP pulp. Cold soda process also gave a strong pulp. However, brightness was very low (48.1). It had a yellow-orange tint and could not be used in white paper grades where high brightness is required. Subsequent bleaching of this pulp with 1% hydrogen peroxide followed by 1% sodium hydrosulfite increased brightness to only 64.3.

#### Examples 5-7

10 Three runs were made to determine the effect of acidification level prior to refining on brightness and on other pulp properties. Different levels of  $\text{SO}_2$  addition were applied to adjust pulp pH between 6 and 10. Pulps in Examples 6 and 7 were additionally acidified with  $\text{SO}_2$  to pH 5.5 - 6.0 after refining. Otherwise, procedures were identical to those of Example 2.

15 Results were as in the following table (Table II).

TABLE II

Example No.	5	6	7
Number of refiner passes	2	2	2
<u>Chemicals Applied on Chips (bone-dry basis),%:</u>			
5 NaOH	7.0	7.0	7.0
H <sub>2</sub> O <sub>2</sub>	1.5	1.5	1.5
DTPA	0.5	0.5	0.5
<u>Pulp Properties:</u>			
10 pH, immediately after refining	5.9	7.2	10.2
Brightness, Initial	71.6	69.9	65.4
Brightness, Aged *	69.8	68.6	64.4
C.S. Freeness, ml	100	112	155
Bulk, cc/g.	1.80	1.76	1.63
15 Tear Factor	67.5	64.8	64.4
Burst Factor	30.5	31.8	32.2
Tensile, b.l.m.	5900	6120	6030
MIT Fold	34	32	27
TAPPI Opacity	81.1	82.3	82.2
20 Optical Scattering, S	.0402	.0405	.0393

\* Aged for 1 hour in oven at 105°C.

25 In these runs the best brightness was obtained when impregnated chips were fully acidified before refining, to obtain a pulp of pH 5.9. Brightness was lower by 6.2 points when chips were only partially acidified (pH 10.2 after refining). Other pulp properties were not affected by the acidification level to a noticeable degree.

Examples 8-13

5 Six runs were made on Sunds Defibrator pilot plant unit containing Defibrator 300 CD disc refiner for primary refining and Raffinator RO 20 disc refiner for secondary refining, both connected  
10 in series by a screw conveyor. Aspen chips were presteamed with low pressure steam in a hopper and then were compressed by a feed screw in a continuous operation. Feed screw delivered compressed chips to a digester containing two chambers. The first chamber was used for chip impregnation and was partially filled with liquor. The  
15 second one served as a retention vessel. Both chambers were under atmospheric pressure. Impregnation chemicals were continuously injected into the first chamber where the compressed chips were submerged for about 10 minutes. Impregnated chips were lifted out from the first chamber and moved into the second chamber for  
20 steeping. From there the chips were screw fed into the primary refiner, followed by secondary refining in Raffinator RO 20. Chip retention time between impregnation and refining was about 20 minutes. Two variables were explored in these 6 runs: peroxide addition level (0.55 to 2.04%) and the location of acidification step (immediately before versus after refining). Acidification after refining was done within 3 minutes after collecting the pulp from the secondary refiner. Sodium bisulfite solution was used for acidification in all 6 runs. Results were as follows:



TABLE III

Example No.	8	9	10	11	12	13
<u>Chemicals Applied in Impregnation (% on bone-dry chips):</u>						
5 NaOH	6.90	6.90	6.77	6.77	6.86	6.86
H <sub>2</sub> O <sub>2</sub>	0.55	0.55	1.06	1.06	2.04	2.04
DTPA	0.5	0.5	0.5	0.5	0.5	0.5
Acidification Point	After Ref.	Before Ref.	After Ref.	Before Ref.	After Ref.	Before Ref.
Consistency during refining, %:						
10 Primary refiner	12.0	9.4	13.5	12.4	14.1	14.8
Secondary refiner	10.2	8.4	12.1	12.6	12.2	14.1
pH, secondary refiner	11.9	6.4	11.4	6.5	11.4	6.2
Brightness, Initial	58.0	64.0	63.2	68.5	70.7	73.8
Brightness, Aged *	56.3	62.1	61.1	66.1	68.9	71.5
15 Canadian Standard Freeness, ml:						
Before latency removal	60	80	80	70	75	70
After latency removal	35	40	60	20	45	10
Handsheet Properties After Latency Removal:						
20 Bulk, cc/g	1.45	1.75	1.52	1.66	1.50	1.59
Tear Factor	55	56	54	55	55	55
Burst Factor	41.0	31.8	38.9	38.2	42.6	40.1
Tensile, b.l.m.	7497	6292	7071	7053	7266	7075
MIT Fold	136	30	77	72	92	55
TAPPI Opacity	83.5	88.9	82.9	84.7	79.3	82.5
25 Optical Scattering, S	.0384	.0492	.0395	.0440	.0376	.0417

\* Aged for 1 hour in oven at 105°C.

According to these data, significant gains in pulp brightness were achieved by acidifying before refining according to the techniques of this invention, and not after refining which has been the accepted way in prior art. Other advantages of acidification before refining is higher opacity and higher bulk, two very desirable pulp properties in the manufacture of printing papers. Pulp strength properties were comparable for runs made with acidification before versus after refining. The only exception was pulp in Example 9, which was somewhat weaker. However, even this pulp is as strong or stronger than some chemical hardwood market pulps. The apparent reason for lower strength in Example 9 is lower consistency during refining (9.4 - 8.4% versus 12.0 - 10.2% in Example 8). This low consistency was discovered only after the runs were completed.

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